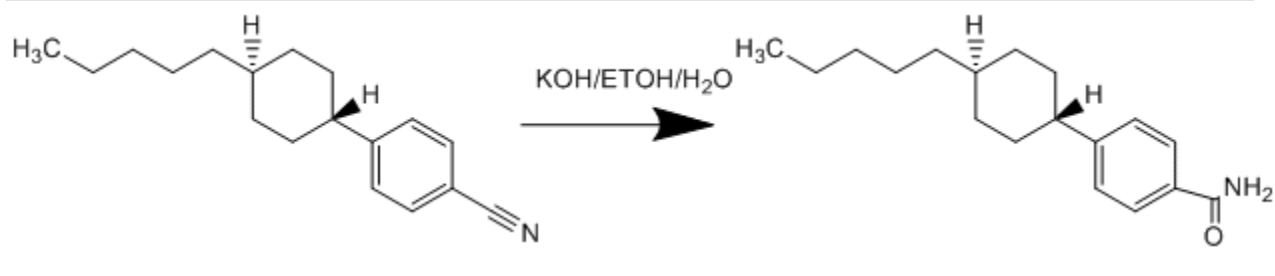


Partial hydrolysis of benzonitriles; 4-(trans-4-Pentylcyclohexyl)benzamide

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Chemicals Used

4-(Trans-4-pentylcyclohexyl)-benzonitrile (Fluorochem), 300840

Potassium hydroxide, pellets, Sigma Aldrich, 85+%, 22,147-3

Ethyl alcohol, 190 proof, Sigma Aldrich, 95+%, 49,351-1

Silicon carbide (boiling stones), Sigma Aldrich, 400 mesh, 35,739-1

Procedure

A 200 ml round bottom one neck flask equipped with a heating mantle and magnetic stirrer was charged with 60 ml of 190 proof ethanol. Stirring was commenced and 4g of potassium hydroxide pellets were added slowly over ~ 15minutes and the mixture further stirred until all pellets had dissolved. Then 3.09g (12 mmole) of 4-(trans-4-pentylcyclohexyl)-benzonitrile was added and the mixture stirred until homogeneous. The solution was then diluted with 20 ml of distilled or deionized water, two silicon carbide boiling stones added, and a water condenser attached. Heating was commenced and the solution taken to full reflux for ~12 hours (overnight). During this period a copious white precipitate of amide separated from the solution. The solution was allowed to cool to room temperature and the precipitate collected on a Buchner funnel by suction filtration. The walls of the flask were washed twice with 10ml water portions which were in turn filtered with the Buchner funnel. Boiling stones and stirring bar were mechanically removed with a spatula. The white solid was transferred to a large watch glass and air dried overnight yielding 2.9g (88%) of amide. The material was analytically pure, required no further purification, and can be used directly in subsequent reactions.

Author's Comments

Potassium hydroxide pellets are very irritating and hygroscopic. Wear latex gloves and weigh quickly or in a dry box. By entirely analogous procedures other benzonitriles were hydrolyzed to amides in high yield with no detectable carboxylic acids from full hydrolysis. See primary and secondary references. Examples include:

p-(trans-4-Propylcyclohexyl)benzamide, m.p. 256-57°.

p-(trans-4-Heptylcyclohexyl)benzamide, m.p. 218-19°.

Data

m.p. 234-5°C

I.r. (Dichloromethane)) 3300,3010,2970,2950,1640 cm⁻¹

Analysis: Calculated for C₁₈H₂₇NO: C, 79.06 H, 9.96 N, 5.12

Found: C,79.36 H,10.04 N, 5.07

Lead Reference

John H. MacMillan and Mortimer M. Labes, "Low Transition Temperature Liquid Crystalline Amines Incorporating the Trans-1,4-Cyclohexane Ring System", Molecular Crystals and Liquid Crystals, Vol. 55, p 61, (1979). DOI: [dx.doi.org/10.1080/00268947908069791](https://doi.org/10.1080/00268947908069791)

Other References

John H. MacMillan and Mortimer M. Labes, "Low Transition Temperature Liquid Crystalline Amines Incorporating the Biphenyl Ring System", Mol. Crystals and Liquid Crystals Letters, Vol. 56, p51, (1979). DOI: Link: <http://dx.doi.org/10.1080/01406567908071966>

John H. MacMillan and Mortimer M. Labes, "Amine Substituted Liquid Crystal Compositions", U.S. Patent 4,293,193, Oct. 6, 1981.

Chemspider deposition: <http://www.chemspider.com/Chemical-Structure.29354290.html>

Keywords: addition, amides, benzamides, benzonitriles, carboxylic acids, cyanides, hydrolysis, hydroxylation, nitriles